

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants : Pramod K. Arora and Brij P. Singh
Serial No. : Unknown
Filed : Unknown
Title : COMPOSITION WITH FILM FORMING
ALKYLSILSESQUIOXANE POLYMER AND
METHOD FOR APPLYING HYDROPHOBIC
FILMS TO SURFACES

Attorney Docket No. : 495263010035

I hereby certify that this correspondence
is being deposited today with the United
States Postal Service as first class mail in
an envelope addressed to: Assistant
Commissioner for Patents Washington,
D.C. 20231

on AUGUST 1, 2001

DECLARATION

H. Duane Switzer declares:

By: H. Duane Switzer

THAT he is an attorney in the State of Ohio and is registered to practice before
the U.S. Patent and Trademark Office;

THAT he sent the attached letter dated June 26, 2001, to Pramod K. Arora with
enclosed copies of formal papers and a patent application for a COMPOSITION WITH FILM
FORMING ALKYLSILSESQUIOXANE POLYMER AND METHOD FOR APPLYING
HYDROPHOBIC FILMS TO SURFACES;

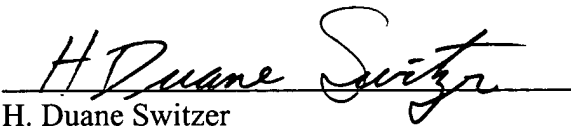
THAT he received the attached email dated June 26, 2001, from Pramod K. Arora
indicating that Mr. Arora would not sign the papers and would return the application;

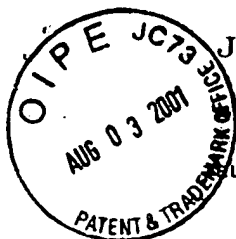
THAT he received the application and unsigned formal papers from Mr. Arora on
July 6, 2001;

THAT Pramod K. Arora contends that he is owed money by nanoFILM, Ltd. for
patents, a contention that is denied by nanoFILM, Ltd.; and

THAT in his opinion, the conduct of Pramod K. Arora constitutes a refusal to sign the application papers.

I HEREBY DECLARE THAT all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.


H. Duane Switzer
Reg. No. 22,431



JONES, DAY, REAVIS & POGUE

NORTH POINT • 901 LAKESIDE AVENUE

CLEVELAND, OHIO 44114-1190

TELEPHONE: 216-586-3939 • FACSIMILE: 216-579-0212

WRITER'S DIRECT NUMBER:

June 26, 2001

216-586-7283

hswitzer@jonesday.com

495263-010-035

CERTIFIED MAIL NO. 7099 3400 0000 7911 0761
RETURN RECEIPT REQUESTED

Mr. Pramod K. Arora
5144 Pinckneya Drive
North Royalton, OH 44133

Re: Proposed U.S. Patent Application For:
COMPOSITION WITH FILM FORMING
ALKYLSILSESQUIOXANE POLYMER
AND METHOD FOR APPLYING
HYDROPHOBIC FILMS TO SURFACES
Our File: 495263-010-035

RECEIVED

AUG 08 2001

OFFICE OF PETITIONS

Dear Mr. Arora:

Enclosed please find a patent application related to the solid state alkylsilsesquioxane polymers that you helped to develop while employed at nanoFILM. Copies of the literature and patents referred to in the patent specification are enclosed for your file.

Please review the application carefully and let me know of any changes that you consider appropriate. If the application is satisfactory, please sign and date all of the formal papers, and return them to me at your earliest convenience.

We would be pleased to answer any questions that you may have, and also remind you that this patent application and its subject matter are covered by the confidentiality agreement that you signed while employed by nanoFILM. Your cooperation is appreciated.

Very truly yours,

H. Duane Switzer

Enclosures:
Application/Drawing
Formal Papers (Declaration & Power of Atty/Assignment)
Prior Art References



"Pramod Arora"
<parora@core.com>

07/03/01 10:15 AM

To: <hswitzer@jonesday.com>
cc:
Subject: patent application 6/26/01

Dear Mr. Switzer,

It is my pleasure talking to you first time in my life. I am sorry that I could not get chance nor given a chance to communicate with you with patent related matters while employed at Nanofilm.

I am also sorry that I could not get back to you earlier as I was out of town for couple of days. Yesterday, my wife told me that you called and it was late to contact. I did received a patent application to review and was asked to sign. I do not have time to go through and to see what you have wrote.

In my earlier patent Brij has added his name in my work, which leads to patent. It seem that Brij and Scott decides whose name should be included in the patent without a inventor or attorney consent. Nanofilm also promised to pay me \$3000 each patent once they get published or awarded. Now they have changed their policy and refused to pay me patent reward. I am seeking legal advice and therefore have no interest to go through this or any future patent to Nanofilm from my work. I do also fully understand that I will keep all subject matters confidential according to my employment agreement with Nanofilm.

If you have any question please reply to this e-mail and I will be very happy to discuss anything you might have.

Thanks,

Pramod Arora

P.S. I will return all documents file (which you sent on 6/26/01) to you tomorrow as it is not appropriate for me keep.



DAE #1

Please type a plus sign (+) inside this box → ☐

PTO/SB/21 (08-00)
Approved for use through 10/31/2002. OMB 0651-0031
U.S. Patent and Trademark Office: U.S. DEPARTMENT OF COMMERCE
Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

TRANSMITTAL FORM <i>(to be used for all correspondence after initial filing)</i>	Application Number	
	Filing Date	
	First Named Inventor	Pramod K. Arora
	Group Art Unit	
	Examiner Name	
Total Number of Pages in This Submission		Attorney Docket Number 495263010035

ENCLOSURES (check all that apply)		
<input checked="" type="checkbox"/> Fee Transmittal Form <input checked="" type="checkbox"/> Fee Attached <input type="checkbox"/> Amendment / Reply <input type="checkbox"/> After Final <input type="checkbox"/> Affidavits/declaration(s) <input type="checkbox"/> Extension of Time Request <input type="checkbox"/> Express Abandonment Request <input type="checkbox"/> Information Disclosure Statement <input type="checkbox"/> Certified Copy of Priority Document(s) <input type="checkbox"/> Response to Missing Parts/ Incomplete Application <input type="checkbox"/> Response to Missing Parts under 37 CFR 1.52 or 1.53	<input type="checkbox"/> Assignment Papers (for an Application) <input type="checkbox"/> Drawing(s) <input type="checkbox"/> Licensing-related Papers <input checked="" type="checkbox"/> Petition Under 37 CFR 1.47 (a) <input type="checkbox"/> Petition to Convert to a Provisional Application <input type="checkbox"/> Power of Attorney, Revocation Change of Correspondence Address <input type="checkbox"/> Terminal Disclaimer <input type="checkbox"/> Request for Refund <input type="checkbox"/> CD, Number of CD(s) _____	<input type="checkbox"/> After Allowance Communication to Group <input type="checkbox"/> Appeal Communication to Board of Appeals and Interferences <input type="checkbox"/> Appeal Communication to Group (Appeal Notice, Brief, Reply Brief) <input type="checkbox"/> Proprietary Information <input type="checkbox"/> Status Letter <input checked="" type="checkbox"/> Other Enclosure(s) (please identify below): DECLARATION OF H. DUANE SWITZER
Remarks		

SIGNATURE OF APPLICANT, ATTORNEY, OR AGENT		
Firm or Individual name	H. Duane Switzer	RECEIVED
Signature	<i>H Duane Switzer</i>	AUG 08 2001
Date	August 1, 2001	OFFICE OF PETITIONS

CERTIFICATE OF MAILING			
I hereby certify that this correspondence is being deposited with the United States Postal Service with sufficient postage as first class mail in an envelope addressed to: Commissioner for Patents, Washington, DC 20231 on this date: 08/01/2001			
Typed or printed name	H. Duane Switzer		
Signature	<i>H Duane Switzer</i>	Date	08/01/2001

Burden Hour Statement: This form is estimated to take 0.2 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231.

PTO/SU/01 (03 01)

Approved through 10/31/2002. OMB 0651-0032

U.S. Patent and Trademark Office, U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION (37 CFR 1.63)

☒ Declaration Submitted with Initial Filing OR ☐ Declaration Submitted after Initial Filing (surcharge (37 CFR 1.16 (e)) required)

Attorney Docket Number	495263010035
First Named Inventor	Pramod K. Arora
COMPLETE IF KNOWN	
Application Number	
Filing Date	
Group Art Unit	
Examiner Name	

As a below named inventor, I hereby declare that:

My residence, mailing address, and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

COMPOSITION WITH FILM FORMING ALKYL SILSESQUOXANE POLYMER
AND METHOD FOR APPLYING HYDROPHOBIC FILMS TO SURFACES

(Title of the Invention)

The specification of which

☒ is attached hereto

OR

☐ was filed on (MM/DD/YYYY)

PETITIONS OFFICE
as United States Application Number or PCT International

Application Number and was amended on (MM/DD/YYYY) (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR 1.56, including for continuation-in-part applications, material information which became available between the filing date of the prior application and the national or PCT international filing date of the continuation-in-part application.

I hereby claim foreign priority benefits under 35 U.S.C. 119(a)-(d) or (f), or 365(b) of any foreign application(s) for patent, inventor's or plant breeder's rights certificate(s), or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent, inventor's or plant breeder's rights certificate(s), or any PCT international application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application Number(s)	Country	Foreign Filing Date (MM/DD/YYYY)	Priority Not Claimed	Certified Copy Attached?	
				YES	NO
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

☐ Additional foreign application numbers are listed on a supplemental priority data sheet PTO/SB/02B attached hereto:

[Page 1 of 2]

Burden Hour Statement: This form is estimated to take 21 minutes to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231.

PTO/SB/01 (03-01)

Approved for use through 10/31/2002, OMB 0851-0032

U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

DECLARATION — Utility or Design Patent ApplicationDirect all correspondence to: ☒ Customer Number OR ☒ Correspondence address belowName **H. Duane Switzer
Jones, Day, Reavis & Pogue**Address **North Point
901 Lakeside Avenue**City **Cleveland**State **OH**ZIP **44114-1190**Country **US**Telephone **216-586-7283**Fax **216-579-0212**

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

NAME OF SOLE OR FIRST INVENTOR: ☒ A petition has been filed for this unsigned inventorGiven Name (first and middle (if any)) **PRAMOD K.**Family Name or Surname **ARORA**

Inventor's Signature

Date

Residence: City **North Royalton**State **OH**Country **US**Citizenship **India**Mailing Address **5144 Pinckney Drive**City **North Royalton**State **OH**ZIP **44133**Country **US**NAME OF SECOND INVENTOR: ☐ A petition has been filed for this unsigned inventorGiven Name (first and middle (if any)) **BRIJ P.**Family Name or Surname **SINGH**

Inventor's Signature

Date **7-02-01**Residence: City **North Royalton**State **OH**Country **US**Citizenship **US**Mailing Address **13010 Morning Star Drive**City **North Royalton**State **OH**ZIP **44133**Country **US**☐ Additional inventors are being named on the _____ supplemental Additional Inventor(s) sheet(s) PTO/SB/02A attached hereto.

Please type a plus sign (+) inside the box



PTO/SB/81 (02-01)

Approved for use through 10/31/2002, OMB 0851-0035

U.S. Patent and Trademark Office, U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

POWER OF ATTORNEY OR AUTHORIZATION OF AGENT

Application Number

Filing Date

First Named Inventor

Pramod K. Arora

Title

Group Art Unit

Examiner Name

Attorney Docket Number

495263010035

I hereby appoint:

☒ Practitioners at Customer Number

24325

OR

☐ Practitioner(s) named below:

Name	Signature



24325

PATENT TRADEMARK OFFICE

as my/our attorney(s) or agent(s) to prosecute the application identified above, and to transact all business in the United States Patent and Trademark Office connected therewith.

Please change the correspondence address for the above-identified application to:

☐ The above-mentioned Customer Number.

OR

☐ Practitioners at Customer Number

Place Customer
Number Bar Code
Label here

☐ Firm or
Individual Name

Address

Address

City

State

Zip

Country

Telephone

Fax

I am the:

☒ Applicant/Inventor.

☐ Assignee of record of the entire interest. See 37 CFR 3.71.
Statement under 37 CFR 3.73(b) is enclosed. (Form PTO/SB/96).

SIGNATURE of Applicant or Assignee of Record

Name

Brij P. Singh

Signature

Date

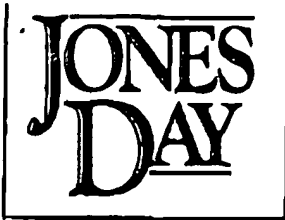
7-02-01

NOTE: Signatures of all the inventors or assignees of record of the entire interest or their representative(s) are required. Submit multiple forms if more than one signature is required, see below.

☒ Total of 2 forms are submitted.

Burden Hour Statement: This form is estimated to take 3 minutes to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231.

FAX RECEIVED
SEP - 5 2001
PETITIONS OFFICE

**Facsimile Transmission**

North Point, 901 Lakeside Avenue • Cleveland, Ohio 44114-1190 • (216) 586-3939
Facsimile: (216) 579-0212

Date: September 5, 2001

Please hand deliver the following facsimile to:

FAX RECEIVED

Name: Mrs. Frances Hicks

Facsimile No.: 703-308-6916

SEP - 5 2001

PETITIONS OFFICE

Company: U.S. Patent and Trademark
Office

Number of Pages (including this page): 4

Telephone No.: 703-305-8680

From: H. Duane Switzer

Send copies to:

Direct Telephone No.: (216) 586-7283

JP No.: 992104

☐ Copies distributed _____
Operator's Initials

CAM No.: 495263010035

NOTICE: This communication is intended to be confidential to the person to whom it is addressed, and it is subject to copyright protection. If you are not the intended recipient or the agent of the intended recipient or if you are unable to deliver this communication to the intended recipient, please do not read, copy or use this communication or show it to any other person, but notify the sender immediately by telephone at (216) 586-3939 or the direct telephone number noted above.

Message:

RE: SERIAL NO. 09/887,661

Please call us immediately at (216) 586-3939 if the facsimile you receive is incomplete or illegible. Please ask for the facsimile operator.

Jones Day, Rivis & Pogue

Atlanta, Brussels, Chicago, Cleveland, Columbus, Dallas, Frankfurt, Geneva, Hong Kong, Irvine, London, Los Angeles,
New Delhi (an Associate Firm), New York, Paris, Pittsburgh, Riyadh, Shanghai, Sydney, Taipei, Tokyo, Washington



JONES, DAY, REAVIS & POGUE

NORTH POINT • 901 LAKESIDE AVENUE

CLEVELAND, OHIO 44114-1190

TELEPHONE: 216-586-3939 • FACSIMILE: 216-579-0212

WRITER'S DIRECT NUMBER:

June 26, 2001

216-586-7283

hswitzer@jonesday.com

495263-010-035

CERTIFIED MAIL NO. 7099 3400 0000 7911 0761
RETURN RECEIPT REQUESTED

Mr. Pramod K. Arora
5144 Pinckney Drive
North Royalton, OH 44133

Re: Proposed U.S. Patent Application For:
COMPOSITION WITH FILM FORMING
ALKYLSILSESQUIOXANE POLYMER
AND METHOD FOR APPLYING
HYDROPHOBIC FILMS TO SURFACES
Our File: 495263-010-035

RECEIVED
AUG 08 2001
OFFICE OF PETITIONS

Dear Mr. Arora:

Enclosed please find a patent application related to the solid state alkylsilsesquioxane polymers that you helped to develop while employed at nanoFILM. Copies of the literature and patents referred to in the patent specification are enclosed for your file.

Please review the application carefully and let me know of any changes that you consider appropriate. If the application is satisfactory, please sign and date all of the formal papers, and return them to me at your earliest convenience.

We would be pleased to answer any questions that you may have, and also remind you that this patent application and its subject matter are covered by the confidentiality agreement that you signed while employed by nanoFILM. Your cooperation is appreciated.

Very truly yours,

H. Duane Switzer

Enclosures:
Application/Drawing
Formal Papers (Declaration & Power of Atty/Assignment)
Prior Art References

**COMPOSITION WITH FILM FORMING ALKYL SILSESQUOXANE
POLYMER AND METHOD FOR APPLYING HYDROPHOBIC
FILMS TO SURFACES**

RELATED APPLICATIONS

5 This application claims subject matter disclosed in U.S.
provisional application Serial No. 60/241,504 filed October 18, 2000, the
benefit of the filing date of which is hereby claimed.

BACKGROUND OF THE INVENTION

10 This application relates to the art of film forming compositions
and to methods for applying films to substrates. The invention is particularly
applicable to film forming compositions that contain solid state
alkylsilsesquioxane polymers and to methods for applying such polymers to
substrates, and will be described with particular reference thereto. However,
15 it will be appreciated that the application has broader aspects and that at least
certain features can be used with other polymers and methods.

 Polymerizable amphiphilic molecules having the intrinsic
ability to self-assemble into a thin film are well known in both solution phase
and gas phase. By way of example, descriptions of such materials and their
20 ability to form thin films are contained in: W.C. Bigelow et al, J. Colloid.
Sci., 1,513-538 (1946); L.H. Lee, J. Colloid. & Interface Sci., 27, 751-760
(1968); E.E. Polymeropoulos et al, J. Chem. Phys., 69, 1836-1847 (1978); J.
Sagiv, U.S. Patent No. 4,539,061; J. Phys. Chem. 70, 2937 (1966); Trans.
Faraday. Soc., 63, 2549 (1967); J. Phys. Chem., 73, 2372 (1969); Langmuir, 7,
25 923 (1991); Langmuir, 9, 3518 (1993) and Langmuir, 13, 1877 (1997).

Disclosures of molecular beam deposition of coatings on substrates are found in the following U.S. patents: 4,001,858; 4,181,544; 4,330,360; 4,681,773; 4,800,100; and 5,064,520. The disclosures of these publications and patents are hereby incorporated herein by reference. Compositions and methods for
5 applying hydrophobic ultra thin films of self-assembling amphiphilic molecules to substrates are described in commonly assigned U.S. Patent Nos. 5,078,791; 5,166,000; 5,173,365; 5,204,126; 5,219,654; 5,300,561; 5,766,698; and 5,897,918. The disclosures of these patents are hereby incorporated herein by reference.

10 Use of the compositions and methods disclosed in the above literature and patents typically results in the formation of a mono-layer thin film on a substrate surface. Inter-molecular interactions in both solution phase and gas phase under a low vacuum make it difficult to use these compositions and methods to form multi-layer films. In addition, the use of these
15 compositions requires cleaning of the substrate surface and/or the vacuum chamber after formation of the film.

Compositions and methods disclosed in the above literature and patents are very sensitive to moisture, and require special packaging, handling and processing. These prior art processes also expose the entire substrate
20 surface to the film forming substance and result in a film over the entire surface of the substrate. There is no choice of selecting a certain substrate surface or shape for film formation other than by masking.

In the compositions and methods disclosed in the above literature and patents, highly reactive self-assembling amphiphilic monomer
25 substances are used to form the films. It would be desirable if these

monomers could be partially polymerized to reduce their high reactivity to moisture while still being capable of acting as self-assembling amphiphilic molecules to form thin films.

Compositions with organic polymer molecules and self-assembling amphiphilic polymer substances for use in forming multi-layer thin films have been reported in the literature. By way of example, silsesquioxanes made from different monomer silanes and alkylsilanes are disclosed in Chem. Rev., 95 1431-1442 (1995) and Chem. Rev., 95, 1409-1430 (1995), and references cited therein and in J. Am. Chem. Soc., 119, 3135-3143 (1997). The disclosures of these publications are hereby incorporated herein by reference.

Methods for applying multi-layer thin films of organic polymers and self-assembling amphiphilic polymer substances inside ultra high vacuum chambers are known in the fields of optoelectronics, flat panel displays, thin film transistors and lasers as disclosed in J. Am. Chem. Soc., 120, 8563-8564 (1998) and Chem. Rev., 97, 1793-1896 (1997), and references cited therein. The disclosures of these publications are hereby incorporated herein by reference.

Use of the above methods and compositions requires the use of materials having extremely high purity. Therefore, a very complicated purification procedure is required that includes the use of a vacuum chamber at an ultra high vacuum of 1×10^{-7} to 1×10^{-11} torr.

It would be desirable to have a process and composition for use in applying hydrophobic thin films of self-assembling amphiphilic polymer substances to surfaces in a manner that is very fast, efficient and cost

effective. It also would be desirable to have a process that is capable of coating only one surface at a time with a film of controlled thickness. It also would be desirable to have a process that could be used at a much lower vacuum than the ultra high vacuum mentioned in the previous references. It
5 also would be desirable to have a process where cleaning of the excess coating material inside the vacuum chamber automatically takes place during the coating process. It would be desirable to have a coating composition of self-assembling amphiphilic polymer substances that is easy to handle and use. It also would be desirable to have a composition that is very stable at room
10 temperature and humidity, and does not require special protection from temperature or moisture. It would be yet another desirable characteristic to have a composition and process that is user friendly and environmentally safe. It would be another desirable characteristic to have a composition and process in which a single component material of very high purity is not required. It
15 further would be desirable to have a coating composition that is easy to dispose of after it has been used.

SUMMARY OF THE INVENTION

In accordance with the present application a stable solid state
20 coating composition includes a solid state film forming polymer having self-assembling amphiphilic molecules. In one arrangement, the film forming polymers are alkylsilsesquioxanes which are prepared in accordance with known procedures, such as disclosed in J. Am. Chem. Soc., 119, 3135-3143 (1997), the disclosure of which is hereby incorporated herein by reference.

A pure film forming substance in accordance with the present application evaporates very rapidly when heated and this makes it difficult to control the thickness of a film that is formed by the evaporated molecules. Therefore, the film forming substance preferably is mixed with an inert
5 carrier, such as a metal oxide, that is stable at high temperatures and does not react with moisture or with the film forming substance.

The composition of film forming polymer powder mixed with a metal oxide powder is compressed into a tablet or compressed into a metal cup. The film forming polymer preferably is 10-50% by weight of the
10 composition.

The amount of film forming substance in the composition that is compressed into a tablet or compressed into a metal cup usually is in the range of 0.5 to 5.0 grams, and more preferably 0.5 to 1.0 grams. Obviously, larger or smaller amounts may be used for some purposes.

15 When a metal cup is used and packed with compressed composition according to the present application, the volume of the cup usually is 0.5 to 2.0 milliliters. Obviously, other sizes may be used for some purposes.

A substrate is coated with a thin film of amphiphilic molecules
20 in accordance with the present application by placing the composition of the present application in a vacuum chamber with a substrate to be coated. A high vacuum of 1×10^{-4} to 1×10^{-6} torr is established and maintained within the vacuum chamber during the coating process. When the desired vacuum is established, the composition is heated within the vacuum chamber to
25 evaporate the film forming substance from a solid state to a vapor state by

sublimation. The evaporated material forms a molecular beam of amphiphilic molecules that settle on the substrate surface and self-assemble into a continuous thin film that bonds to the substrate surface. The thickness of the film is controlled by the evaporation rate of the film forming substance and
5 time.

A variety of different substrate materials can be coated with thin films of amphiphilic polymer molecules by using the method and composition of the present application. Suitable substrate materials include, but are not necessarily limited to, glass, ceramic, porcelain, plastics, glass or
10 plastic lenses, glass slides, sun glasses, safety glasses, precision optical parts, lenses with anti-reflective coatings, or flat sheets or other surfaces, and certain polished metal surfaces such as silicon, aluminum, germanium, chromium, titanium and zirconium.

It is a principal object of the present invention to provide an
15 improved coating composition that contains a solid state film forming substance of amphiphilic molecules for use in providing hydrophobic thin films on substrate surfaces.

It is also a principal object of the invention to provide an improved method for providing hydrophobic thin films on substrate surfaces.

20 It is another object of the invention to provide a method that permits coating of substrate surfaces one side at a time.

It is a further object of the invention to provide a method that can be used to provide substrate surfaces with multi-layer self-assembled films of controlled thickness.

It is also an object of the invention to provide a method that does not require an ultra high vacuum.

It is an additional object of the invention to provide a composition of the type described that is easy to handle, transport and use.

5 It is another object of the invention to provide such a composition that is very stable at normal temperature and humidity.

It is yet another object of the invention to provide a method and composition that is user friendly and environmentally safe.

10 It is also an object of the invention to provide a method wherein excess coating material is removed from the vacuum chamber during the coating process.

It is also an object of the present invention to provide a coating composition that is easy and safe to dispose of.

15 It is also an object of the invention to use a mixture of amphiphilic polymers to create good hydrophobic films on surfaces.

BRIEF DESCRIPTION OF THE DRAWING

The drawing is a diagrammatic illustration of a vacuum chamber in which the coating method of the present application as carried out.

20

DESCRIPTION OF REPRESENTATIVE EMBODIMENTS

It will be understood that the explanations provided herein are for purposes of disclosing representative embodiments of the invention and not for purposes of limiting same.

As used in the context of this application, a film forming substance is one containing amphiphilic polymeric molecules that are capable of self-assembly on a substrate surface and of bonding thereto by virtue of the high affinity that the polar groups in the polymeric molecules have for the polar groups on the substrate surface. An amphiphile contains a polar region and a non-polar region, and amphiphiles that can be used to form film in accordance with the present application include, but are not necessarily limited to, the following:

The polar segment of the amphiphile can be a carboxylic acid, alcohols, thiols, amides, primary, secondary, tertiary amines, silane derivatives and sulfonates.

The non-polar or apolar component typically consists mainly of alkyl and alkyl ether or fluorinated alkyl and alkyl ether groups. The alkyl chain also may have other polymerizable moieties in it.

In one arrangement, the film forming substance is prepared by the hydrolysis and polymerization of monomers using known procedures as disclosed in the aforementioned literature. The typical monomers used in the present application consist essentially of R_mSiX_n where the non-polar R is a substituted silane or siloxane, or an alkyl, a per-fluorinated alkyl, an alkyl ether, or a per-fluorinated alkyl ether group of 6-20 carbon atoms and most preferably 10-20 carbon atoms, where X is selected from the group consisting of halogens, hydroxy, alkoxy and acetoxy groups, and where m is 1-3, n is 1-3 and $m+n$ equal 4.

In another example, the monomer used to make the stable solid state film forming alkylsilsesquioxane polymer is R_mSiX_n , where R is C_{18} , X is an ethoxy group, m is 1-3, n is 1-3 and $m+n$ equal 4.

By way of example, octadecyltrichlorosilane is used to make a
5 stable solid state film forming amphiphilic polymer substance.

Octadecyltrichlorosilane is added dropwise to a stoichiometric excess of water held at about $5^{\circ}C$ and with good stirring. In the beginning, the material hydrolyzes and suspends in the water solution. After about 15 minutes it rises to the top of the water as a white flaky material and is left standing for 30-45
10 minutes. The precipitate is collected by suction filtration, thoroughly washed with water to remove residual hydrochloric acid, and dried under a vacuum at room temperature which usually is in the range of $18-32^{\circ}C$. A mixture of different siloxane polymers is obtained as mentioned in the literature, and the polymers still have some unreacted active hydroxy groups.

15 The white flaky material is then heated at $160-180^{\circ}C$ for 1 hour at a vacuum not lower than 1×10^{-2} to 5×10^{-2} torr. A lower vacuum would be 1×10^{-1} torr, and higher vacuums would be 1×10^{-3} to 1×10^{-7} . During this process, most of the residual water and possibly the water between different polymer layers is removed. This step is necessary to obtain a
20 polymer that provides a very uniform film deposition rate in the high vacuum process. Without this dehydration step, the deposition rate is not constant due to the release of excess water from the substance during the coating process. However, it is not desirable to obtain nearly 100% dehydration as might be obtained if the vacuum and/or temperature are too high, or if the dehydration
25 time is too long.

The cooled solid polymer material is crushed to a fine powder and mixed with an inert binder such as a metal oxide powder to obtain a homogeneous mixture. Titanium dioxide powder, such as P25 available from Degussa Corporation, is a suitable binder. Other binders that may be useable
5 include silica and alumina. The important characteristic of the binder is that it should be one that does not react to moisture or with the film forming substance, and is stable at high temperatures of 300°C and greater so that it does not evaporate when the composition is heated to evaporate the film forming polymer by sublimation.

10 The thoroughly mixed polymer powder and metal oxide powder are combined so that the polymer powder is 10-50% by weight of the composition, more preferably 20-40% by weight of the composition, and most preferably 25-30% by weight of the composition. The homogeneous mixture is compressed into a tablet or placed in a container such as a small metal cup
15 and compressed therein. The tablet or the homogeneous mixture compressed into the cup is used inside the vacuum chamber for coating substrates with thin films. The metal cup may be of such metals as copper, aluminum and tin, but is not necessarily limited thereto.

In the method of the present application, substrates to be coated
20 are placed inside the vacuum chamber, along with the composition of the present application, and a high vacuum of 1×10^{-4} to 1×10^{-6} torr is established inside the vacuum chamber. The substrate preferably is rotated while the composition is heated to evaporate the solid state film forming substance by sublimation. This establishes a molecular beam of amphiphilic
25 molecules which settle on the substrate surface and attach or bond thereto by

way of covalent bonding, hydrogen bonding and/or van der Waals forces while self-assembling into a continuous thin film. The rate of the deposition is set at 0.1-1.0 nm/sec by controlling the heat and evaporation rate, and is monitored by the use of an optical balance located within the vacuum chamber or by other deposition rate monitors such as a vacuum microbalance or quartz-crystal oscillator. A multi-layer thin film having a uniform thickness of 3-100 nm may be obtained. When the film has reached the desired thickness, heating of the coating composition is stopped and the chamber is vented so that the coated substrates can be removed. This method provides a very uniform hydrophobic thin film on substrate surfaces.

The method of the present application may be used to provide a thin film over other coatings such as anti-reflective coatings and mirror coatings. For example, the composition of the present application may be placed within a vacuum chamber at the same time as a composition for forming an anti-reflective coating or a mirror coating. The anti-reflective or mirror coating is first evaporated to provide the substrates, such as lenses, with an anti-reflective or mirror coating. The composition of the present application then is evaporated to provide a continuous thin film over the anti-reflective coating or mirror coating. Thus, the substrate is sequentially coated with different films without removing it from the vacuum chamber.

A vacuum chamber used to practice the method of the present application may be of the type manufactured by Satis, Denton or Zeiss for use in depositing anti-reflective coatings on lenses. The metal cup containing the composition may be heated with an electron beam gun, a resistance heater, an induction heater or another heat source. The tablet may be placed in a

crucible within the vacuum chamber and similarly heated to evaporate the solid state film forming substance by sublimation. Preferably, neither the substrate nor the vacuum chamber are heated before or during the deposition process, and the only heat produced within the chamber is that used to
5 evaporate the film forming substance. Thus, the temperature within the vacuum chamber during the entire process normally is well below 100°C.

The drawing shows a typical vacuum chamber A having a suitable door for providing access to the interior 10 thereof in a known manner. A conduit 12 communicating with the vacuum chamber interior 10 is
10 connected with a vacuum pump for establishing and maintaining a desirable vacuum within the vacuum chamber.

A rotatable shaft 14 extends through a packing gland 16 to interior 10 of chamber A and has a mechanical gripping device 18 thereon for gripping the outer periphery of a substrate B. Any of the known mechanical
15 clips and holders may be used for holding one or more substrates to be coated, as well as vacuum holders in which one or more substrates, such as lenses, are held to a rotatable support by a vacuum acting on the rear surfaces of the substrates, the vacuum being applied through a hollow shaft 14 and a plurality of spaced-apart ports in a hollow disc holder.

20 A metal cup 20 containing the composition of the present application, or a crucible holding a composition tablet, is positioned on a support 22 having a suitable heater associated therewith for heating the composition to a temperature of 100-300°C and more preferably 150-200°C. After establishing a desirable vacuum of 1×10^{-4} to 1×10^{-6} torr in the vacuum
25 chamber, the heater is energized and the solid state film forming substance in

the composition evaporates by sublimation to form a molecular beam 24 of amphiphilic molecules which settle on substrate surface 26 that faces toward the source 20 of the molecular beam. The opposite surface 28 of the substrate B is not coated with the film forming substance. The amphiphilic molecules
5 settle on substrate surface 26 and bond thereto as by covalent bonding, hydrogen bonding and/or van der Waals forces while simultaneously self-assembling into a continuous thin film. The operation is continued for a period required to form a desired film thickness. the chamber is then vented and the coated substrates are removed.

10 Controlling the heat source used to evaporate the film forming substance controls the evaporation rate which in turn controls the deposition rate of the amphiphilic molecules on the substrate surface. The percent of film forming substance in the composition also may be varied to vary the evaporation and deposition rates.

15 The substrate preferably is located between the source of the molecular beam and the vacuum port with the surface to be coated facing toward the molecular beam so that the molecules in the beam engage the substrate surface as they travel toward the vacuum port. The substrate is rotated at a rate of one revolution per 1-10 seconds (6-60 revolutions per
20 minute), and more preferably one revolution per 2-5 seconds (12-30 revolutions per minute). Removal of excess coating material from the substrate or the vacuum chamber is not necessary. The used composition cup or tablet is simply removed and replaced for coating a new batch of substrates.

By way of example, the film forming substance that is made
25 from R_mSiX_n may be one in which R is an alkyl chain containing 12 carbon

atoms and X is Cl. R also may be a per fluorinated alkyl group containing 12 carbon atoms. R also may be a per fluorinated alkyl chain with X being chloride. R also may be an alkyl chain with 16 carbon atoms. A mixture of different monomers containing alkyl chains ranging from 6 to 12 carbon atoms
5 may be hydrolyzed to provide a film forming substance that is a mixture of different materials that are then mixed with an inert binder powder. Durable and uniform films with excellent hydrophobic properties are obtained using such materials.

Although the invention has been shown and described with
10 reference to representative embodiments, it is obvious that alterations and modifications will occur to others skilled in the art upon reading and understanding of this application. Therefore, it is to be understood that the invention may be practiced otherwise than as specifically described herein while remaining within the scope of the claims.

15

20

25

We Claim:

1. A composition that includes a solid state film forming alkylsilsesquioxane polymer and an inert binder.
- 5 2. The composition of claim 1 wherein said solid state film forming alkylsilsesquioxane polymer comprises 10-50% by weight of said composition.
3. The composition of claim 1 pressed into a tablet.
- 10 4. The composition of claim 1 pressed into a metal cup.
5. The composition of claim 1 wherein said solid state film forming alkylsilsesquioxane polymer is derived from R_mSiX_n where the
15 non-polar R is a substituted silane or siloxane, an alkyl, a per-fluorinated alkyl, an alkyl ether, or a per-fluorinated alkyl ether group of 6-20 carbon atoms and most preferably 10-20 carbon atoms, where X is selected from the group consisting of halogens, hydroxy, alkoxy and acetoxy groups, and where m is 1-3, n is 1-3 and $m+n$ equal 4.
- 20 6. The composition of claim 1 wherein said solid state film forming alkylsilsesquioxane polymer is derived from R_mSiX_n , where R is C_{18} , X is an ethoxy group, m is 1-3, n is 1-3 and $m+n$ equal 4.

7. The composition of claim 1 wherein said solid state film forming alkylsilsesquioxane polymer is derived from alkylsilanes.

8. The composition of claim 1 wherein said solid state film forming alkylsilsesquioxane polymer is derived from R_mSiX_n where R is an alkyl and alkyl ether or a fluorinated alkyl and fluorinated alkyl ether chain containing C6-C20, where X is Cl, Br, I, an alkoxy group or an acetoxy group, and where m is 1-3, n is 1-3 and $m+n$ equal 4.

9. The composition of claim 1 wherein said solid state film forming alkylsilsesquioxane is derived from octadecyltrichlorosilane.

10. The composition of claim 1 wherein said binder includes one or more of titanium dioxide, silica and alumina.

11. The composition of claim 1 wherein said binder comprises metal oxide powder.

12. A composition containing a metal oxide powder and 10-50% by weight of solid state film forming alkylsilsesquioxane polymer powder.

13. The composition of claim 12 wherein said composition is compressed into a tablet.

14. The composition of claim 12 wherein said composition is compressed into a metal cup.

15. A composition containing a metal oxide powder and 10-50% by weight of a solid state film forming substance having amphiphilic molecules that are capable of self-assembly into a thin film on a substrate surface.

16. A method of coating substrate surfaces with a hydrophobic thin film of amphiphilic molecules comprising the steps of positioning a substrate and a solid state film forming substance of amphiphilic molecules within a vacuum chamber, evaporating the film forming substance to form a molecular beam of amphiphilic molecules, and allowing the amphiphilic molecules in the molecular beam to settle on the substrate surface and self-assemble thereon into a hydrophobic thin film.

17. The method of claim 16 including the step of rotating said substrate while said amphiphilic molecules in said molecular beam settle thereon within said vacuum chamber.

20

18. The method of claim 16 including the step of maintaining the temperature within said vacuum chamber at less than 100°C.

19. The method of claim 16 wherein said step of evaporating is carried out to provide a film formation on the substrate surface at a rate of 0.1-1.0 nanometers of film thickness per second.

5 20. The method of claim 19 wherein the film formation rate is 0.4-0.6 nanometers of film thickness per second.

21. The method of claim 16 wherein said method is carried out for a time to provide the substrate with a film having a thickness of 3-100
10 nanometers.

22. The method of claim 21 wherein the method is carried out for a time to provide the substrate with a film having a thickness of 6-15
15 nanometers.

23. The method of claim 16 including the step of maintaining the vacuum chamber at a vacuum of 1×10^{-4} to 1×10^{-6} torr.

24. The method of claim 16 wherein the step of positioning
20 a solid state film forming substance of amphiphilic molecules within a vacuum chamber is carried out by positioning within the vacuum chamber a composition that includes a mixture of an inert powder and a powdered film forming substance of amphiphilic molecules.

25. The method of claim 24 wherein the step of positioning a composition in the chamber is carried out by positioning the composition in the form of a compressed tablet.

5 26. The method of claim 24 wherein the step of positioning a composition in the chamber is carried out by positioning the composition compressed within a metal cup.

10 27. The method of claim 24 wherein the step of positioning a composition is carried out positioning a composition that includes a mixture of a metal oxide powder and a powdered film forming substance of amphiphilic molecules.

15 28. The method of claim 27 wherein the step of positioning a composition is carried out by positioning a composition that contains 10-50% by weight of the powdered film forming substance of amphiphilic molecules.

20 29. A method of coating substrate surfaces with a hydrophobic thin film of amphiphilic molecules comprising the steps of positioning within a vacuum chamber a substrate and a solid composition that contains a solid state film forming substance of amphiphilic molecules, heating the composition to evaporate the film forming substance and form a molecular beam of amphiphilic molecules, allowing the amphiphilic
25 molecules in the molecular beam to settle on the substrate surface and self-

assemble thereon into a hydrophobic thin film, and maintaining the temperature within the vacuum chamber below 100°C.

30. The method of claim 29 including the step of
5 maintaining the vacuum chamber at a vacuum of 1×10^{-4} to 1×10^{-6} torr.

31. In a method of producing a solid state film forming
alkylsilsesquioxane polymer of amphiphilic molecules by the hydrolysis and
polymerization of monomers, the step of heating the alkylsilsesquioxane
10 polymer in a vacuum to remove residual water therefrom and provide a
dehydrated product.

32. The method of claim 31 wherein the step of heating in a
vacuum is carried out at a temperature of 160-180°C.
15

33. The method of claim 32 wherein the step of heating in a
vacuum is carried out at a vacuum at least as low as 1×10^{-2} torr.

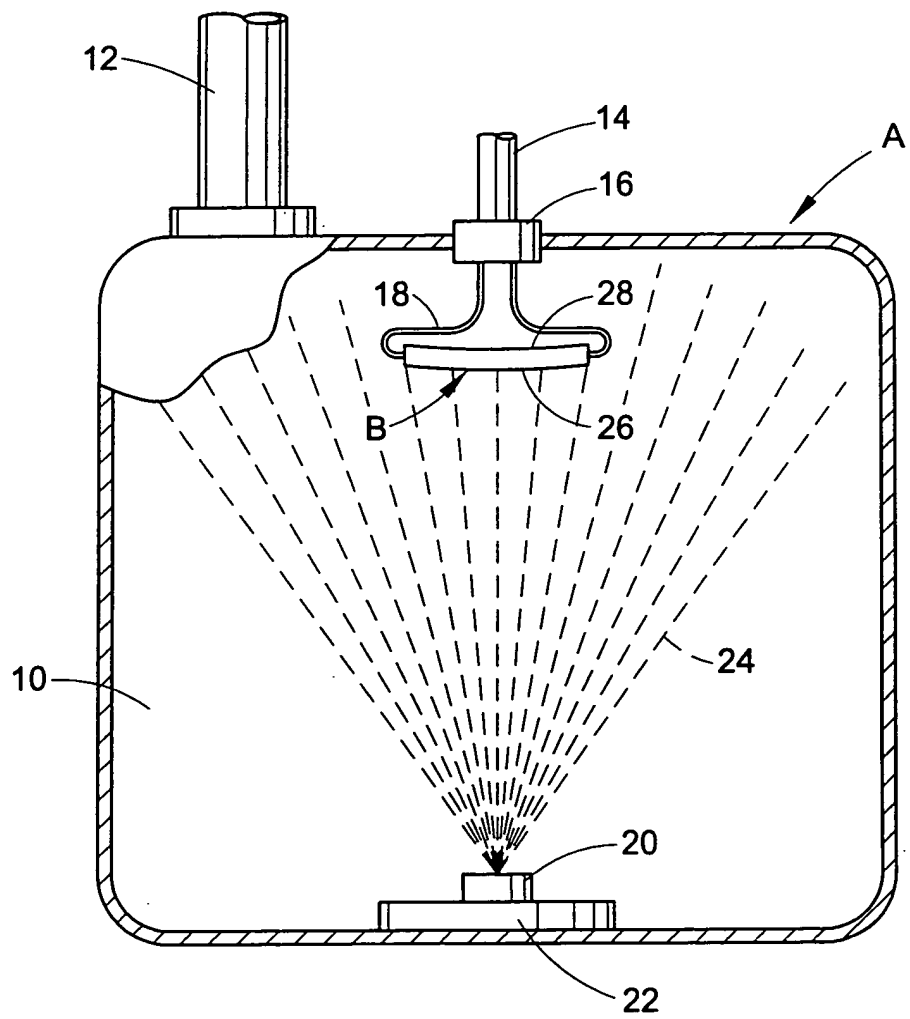
34. The method of claim 33 wherein the step of heating in a
20 vacuum is carried out for at least one hour.

35. The method of claim 31 including the step of crushing
the dehydrated alkylsilsesquioxane polymer product to a fine powder.
25

**COMPOSITION WITH FILM FORMING ALKYLSESQUIOXANE
POLYMER AND METHOD FOR APPLYING HYDROPHOBIC
FILMS TO SURFACES**

ABSTRACT OF THE DISCLOSURE

- 5 A solid composition having a solid state film forming substance mixed with an inert carrier. The composition is heated in a vacuum chamber to evaporate the film forming substance by sublimation to form a molecular beam of amphiphilic molecules which settle on a substrate surface within the chamber and bond thereto while self-assembling into a thin film.



Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

**DECLARATION FOR UTILITY OR
DESIGN
PATENT APPLICATION
(37 CFR 1.63)**

☒ Declaration Submitted with Initial Filing OR ☐ Declaration Submitted after Initial Filing (surcharge (37 CFR 1.16 (e)) required)

Attorney Docket Number 495263010035

First Named Inventor Pramod K. Arora

COMPLETE IF KNOWN

Application Number /

Filing Date

Group Art Unit

Examiner Name

As a below named inventor, I hereby declare that:

My residence, mailing address, and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

COMPOSITION WITH FILM FORMING ALKYL SILSESQUOXANE POLYMER
AND METHOD FOR APPLYING HYDROPHOBIC FILMS TO SURFACES

(Title of the Invention)

the specification of which

☒ is attached hereto

OR

☐ was filed on (MM/DD/YYYY) as United States Application Number or PCT International

Application Number and was amended on (MM/DD/YYYY) (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR 1.56, including for continuation-in-part applications, material information which became available between the filing date of the prior application and the national or PCT international filing date of the continuation-in-part application.

I hereby claim foreign priority benefits under 35 U.S.C. 119(a)-(d) or (f), or 365(b) of any foreign application(s) for patent, inventor's or plant breeder's rights certificate(s), or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent, inventor's or plant breeder's rights certificate(s), or any PCT international application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application Number(s)	Country	Foreign Filing Date (MM/DD/YYYY)	Priority Not Claimed	Certified Copy Attached?	
				YES	NO
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

☐ Additional foreign application numbers are listed on a supplemental priority data sheet PTO/SB/02B attached hereto:

[Page 1 of 2]

Burden Hour Statement: This form is estimated to take 21 minutes to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231.

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

DECLARATION — Utility or Design Patent Application

Direct all correspondence to: <input checked="" type="checkbox"/>		Customer Number or Bar Code Label		OR <input checked="" type="checkbox"/>		Correspondence address below	
Name H. Duane Switzer Jones, Day, Reavis & Pogue							
Address North Point 901 Lakeside Avenue							
City Cleveland				State OH		ZIP 44114-1190	
Country US			Telephone 216-586-7283			Fax 216-579-0212	
I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.							
NAME OF SOLE OR FIRST INVENTOR :				<input type="checkbox"/> A petition has been filed for this unsigned inventor			
Given Name PRAMOD K. (first and middle (if any))				Family Name ARORA or Surname			
Inventor's Signature						Date	
Residence: City North Royalton			State OH		Country US		Citizenship India
Mailing Address 5144 Pinckney Drive							
City North Royalton			State OH		ZIP 44133		Country US
NAME OF SECOND INVENTOR :				<input type="checkbox"/> A petition has been filed for this unsigned inventor			
Given Name BRIJ P. (first and middle (if any))				Family Name SINGH or Surname			
Inventor's Signature						Date	
Residence: City North Royalton			State OH		Country US		Citizenship US
Mailing Address 13010 Morning Star Drive							
City North Royalton			State OH		ZIP 44133		Country US
<input type="checkbox"/> Additional inventors are being named on the ____ supplemental Additional Inventor(s) sheet(s) PTO/SB/02A attached hereto.							

Please type a plus sign (+) inside this box → +

PTO/SB/81 (02-01)

Approved for use through 10/31/2002. OMB 0651-0035

U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it display a valid OMB control number.

POWER OF ATTORNEY OR AUTHORIZATION OF AGENT

Application Number	
Filing Date	
First Named Inventor	Pramod K. Arora
Title	
Group Art Unit	
Examiner Name	
Attorney Docket Number	495263010035

I hereby appoint:

☒ Practitioners at Customer Number

24325

OR

☐ Practitioner(s) named below:

Name	Registration Number

24325

PATENT TRADEMARK OFFICE

as my/our attorney(s) or agent(s) to prosecute the application identified above, and to transact all business in the United States Patent and Trademark Office connected therewith.

Please change the correspondence address for the above-identified application to:

☐ The above-mentioned Customer Number.

OR

☐ Practitioners at Customer Number

Place Customer
Number Bar Code
Label here

☐ Firm or
Individual Name

Address

Address

City

State

Zip

Country

Telephone

Fax

I am the:

☒ Applicant/Inventor.

☐ Assignee of record of the entire interest. See 37 CFR 3.71.
Statement under 37 CFR 3.73(b) is enclosed. (Form PTO/SB/96).

SIGNATURE of Applicant or Assignee of Record

Name	Pramod K. Arora
Signature	X
Date	✓

NOTE: Signatures of all the inventors or assignees of record of the entire interest or their representative(s) are required. Submit multiple forms if more than one signature is required, see below*.

☒ Total of 2 forms are submitted.

Burden Hour Statement: This form is estimated to take 3 minutes to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231.

JOINT

A S S I G N M E N T

In consideration of One Dollar and other good and valuable consideration, receipt of which is hereby acknowledged, I, **PRAMOD K. ARORA**, ("ASSIGNOR"), a citizen of India, residing in North Royalton, Ohio, hereby sell, transfer, set over and assign unto **nanoFILM, Ltd.** ("ASSIGNEE"), an Ohio Limited Liability Company of the State of Ohio, having a principal place of business at 10111 Sweet Valley Drive, Valley View, Ohio 44125-4250, its successors, assigns, nominees, or other legal representatives, the entire worldwide right, title and interest in and to the invention **COMPOSITION WITH FILM FORMING ALKYL SILSESQUOXANE POLYMER AND METHOD FOR APPLYING HYDROPHOBIC FILMS TO SURFACES** invented jointly by me and **BRIJ P. SINGH**, and the application for United States patent therefor, executed concurrently herewith, and all original and reissued patents granted therefor, and all divisions and continuations thereof, including the subject matter of any and all claims which may be obtained in every such patent, and the right to apply for and obtain patents in countries foreign to the United States, and in and to any Letters Patent which may be granted thereon in such foreign countries, and authorize and request the Commissioner of Patents and Trademarks of the United States, and any official of any country or countries foreign to the United States whose duty it is to issue patents on applications as aforesaid, to issue the said Letters Patent to the said ASSIGNEE, its successors, assigns, nominees or other legal representatives, as assignee and owner of the entire interest, and covenant that I have full right to convey the entire interest herein assigned and that I have not executed and will not execute any agreement in conflict herewith, and agree that I will communicate to said ASSIGNEE, its successors, assigns, nominees or other legal representatives, all facts known to me respecting said invention, whenever requested, and testify in any legal proceedings, sign all lawful papers, execute and deliver all divisional, continuing and reissue applications, make lawful papers, execute and deliver all divisional, continuing and reissue applications, make all rightful oaths and do all lawful acts requisite for the application for such divisional, continuing or reissue applications, or the procuring thereof, and that if and when said ASSIGNEE, its successors, assigns, nominees or other legal representatives desire to file a disclaimer relating thereto I will, upon request, sign and deliver all

lawful papers' requisite for the filing of such disclaimer, and I further covenant and agree that I will, at any time upon request, do everything legally possible to aid said ASSIGNEE, its successors, assigns, nominees or other legal representatives, either in its or my own name, to apply for, obtain and enforce proper patent protection in all countries, including priority rights granted to patents in foreign countries according to all the laws and treaties in force, all without further consideration but at the expense of said ASSIGNEE, its successors, assigns, nominees or other legal representatives.

Pramod K. Arora

STATE OF OHIO)
)SS.
COUNTY OF _____)

This _____ day of _____, 2001 before me personally came the above-named Pramod K. Arora, to me personally known as the individual who executed the foregoing Assignment, and who acknowledged to me that he executed the same of his own free will for the purpose therein set forth.

Notary Public

(SEAL)



"Pramod Arora"
<parora@core.com>

To: <hswitzer@jonesday.com>
cc:
Subject: patent application 6/26/01

07/03/01 10:15 AM

B

Dear Mr. Switzer,

It is my pleasure talking to you first time in my life. I am sorry that I could not get chance nor given a chance to communicate with you with patent related matters while employed at Nanofilm.

I am also sorry that I could not get back to you earlier as I was out of town for couple of days. Yesterday, my wife told me that you called and it was late to contact. I did received a patent application to review and was asked to sign. I do not have time to go through and to see what you have wrote.

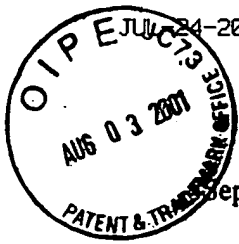
In my earlier patent Brij has added his name in my work, which leads to patent. It seem that Brij and Scott decides whose name should be included in the patent without a inventor or attorney consent. Nanofilm also promised to pay me \$3000 each patent once they get published or awarded. Now they have changed their policy and refused to pay me patent reward. I am seeking legal advice and therefore have no interest to go through this or any future patent to Nanofilm from my work. I do also fully understand that I will keep all subject matters confidential according to my employment agreement with Nanofilm.

If you have any question please reply to this e-mail and I will be very happy to discuss anything you might have.

Thanks,

Pramod Arora

P.S. I will return all documents file (which you sent on 6/26/01) to you tomorrow as it is not appropriate for me keep.



JUL 24-2001 11:30

NANOFILM

2164471137 P.04/04

September 19, 2000

To: Dr. Brij Singh
VP, Technology
NANOFILM

From: Dr. Pramod Arora
Director, Thin Film Technology

Sub: Resignation from employment

Dear Dr. Singh,

Over the years working at NANOFILM was very exiting and challenging and I tried to be a loyal employee. It was a great experience working under your supervisions and some time it was very difficult to perform my duties under very tight schedule without getting an award. I have always enjoyed my profession, no matter where I have worked with true devotion and honesty. I can not fool others or myself. I am a person who always respect peoples and helps them when they needed the most. Last year, I was told that company will provide all the resources and instrumentation to perform my duties and finish a project in a scientific and systematic manner. But due to limited resources and not having proper instrumentation in hand my performance was always judged on the basis of accomplishment of a project.

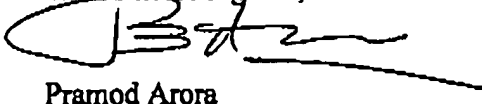
A manager or a company representative should give all the resources and required instrumentation to a project director and let the person do his job in a best possible manner. Project director is required to submit monthly report.

I am submitting my resignation effective immediately. I will be cleaning my office and give all the necessary notebook and papers to you by the end of this week. Therefore, this Friday, September 22, 2000 will be my last day at NANOFILM.

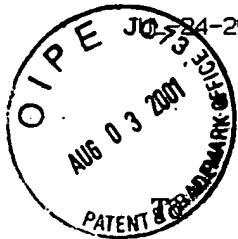
I also understand that I will be rewarded for each patents a sum of \$3000 each once they get approved or published. We have submitted two patents (1998 and 1999) and there are two more to be submitted and these are Plasma Cup and Anti-fog hard coating. Pelisse on car windows, PermaSeal for AR lenses and InkBeGone products should also be rewarded as well if company market them in the next 12 months.

Thank you for your help and co-operation during my stay at NANOFILM. If I can be any helpful to you or anyone while not employed by NANOFILM please never hesitate to contact me at any time.

With sincere regards,



Pramod Arora



D

Pramod Arora

From: Brij Singh

Date: September 22, 2000

RE: Resignation Effective on September 22, 2000

Dear Pramod,

First I want to thank you for 10 years of service to Nanofilm. You have made many contributions, and I wish you the very best in your future endeavors.

In your letter of resignation, you mentioned a potential future cash bonus for two potential patents at a future time when the patents are approved. Nanofilm realizes that there is a possible misunderstanding regarding our current incentive plan in R & D, and we wish to resolve that misunderstanding in a manner that works for both you and the company. The plan with which we are currently operating provides a potential salary increase at the time a new product is commercialized, not a cash bonus. This plan is somewhat different than some of our practices in the past, such as in 1998 and 1999. We would like to resolve this misunderstanding in the following way.

In return for your agreements outlined below, we will continue your salary for two additional pay periods and maintain your enrollment in company benefits. This would result in a normal direct deposit in the amount of your regular two week salary on October 6 and again on October 20. The agreements are as follows.

You agree to fully honor your non-disclosure agreement with the Company. You agree not to compete with our Company in our direct lines of product for a period of one year by working for or consulting with our current customers on our specific products. The specific customers covered by this agreement are Satis Vacuum, Barberini, Pentax Optical, Denton Vacuum, and other current customers for our direct lines of products. Please acknowledge your agreement to these terms by signing a copy of this letter below. Should you wish some time to think about this agreement, it can remain open up to September 26, 2000. After that date it will no longer be valid.

In addition, we want to clarify the purchase of your 1,900 Equity Credits by Nanofilm. Per the Equity Credit plan, Nanofilm will buy back your shares at the current price of \$3.00 per share. The total current value of these shares is \$5,700. You have purchased these shares in 3 purchase transactions for a total cost of \$2,238. As a result, the \$3,462 difference between your cost and the current value is recognized compensation and will be subject to normal payroll taxes. Nanofilm will be paying you \$5,700 minus the tax withholdings required on the recognized compensation portion no later than October 6. You will also be receiving a refund of \$206.25 for withholdings that you authorized for this year.

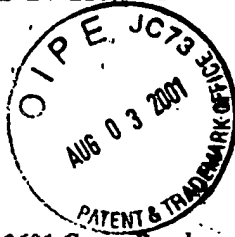
Thanks again for your many years of contribution and good luck in your future.

Sincerely,

Brij Singh
Vice President, Technology

Agreed to by: _____

Date: _____



3681 Green Road
Suite 410
Beachwood, Ohio 44122

CARYN M. GROEDEL
Attorney at Law

Phone: (216) 831-7077
Fax: (216) 831-2135
email: cgroedel@aol.com

July 10, 2001

via facsimile: 216-447-1199

Dr. Scott Rickert
10111 Sweet Valley Drive
Valley View, OH 44125

Dear Dr. Rickert:

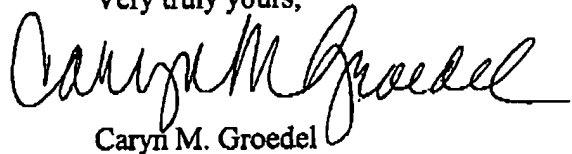
I am writing on behalf of Pramod Arora, who has sought my legal advice and counsel in connection with Nanofilm's non-payment of certain performance incentives he was promised during his employment with Nanofilm.

Specifically, Dr. Arora is entitled to \$1,000 for each patent obtained on his work as well as \$2,000 for each process and product he developed that is sold in the marketplace, for a total of \$3,000 for each patent. Thus far, he was paid \$3,000 for the gel patent. However, he is still owed the \$3,000 bonus for the MgF2 patent obtained in November of 2000, as well as \$3,000 for the Ampoule patent obtained in January 2001, and \$3,000 for the Container patent obtained in March 2001, for a total of \$9,000. Moreover, if the Plasma Cup patent is obtained, which was recently submitted for approval by Nanofilm, Dr. Arora will be entitled to an additional \$3,000.

We have your promises in writing, and are willing to litigate this matter if full payment is not made within two weeks from today.

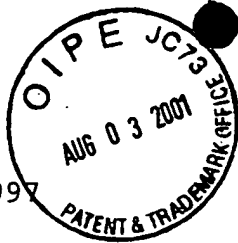
I urge you or your attorney to contact me promptly in an effort to resolve this issue amicably.

Very truly yours,



Caryn M. Groedel

CMG:ji



UNITED STATES DEPARTMENT OF COMMERCE
Patent and Trademark Office
ASSISTANT SECRETARY AND COMMISSIONER
OF PATENTS AND TRADEMARKS
Washington, D.C. 20231

F

APRIL 04, 1997

PTAS

BARBARA E. ARNDT, PH.D.
JONES, DAY, REAVIS & POGUE
NORTH POINT - 901 LAKESIDE AVENUE
CLEVELAND, OH 44114-1190



100351144A

UNITED STATES PATENT AND TRADEMARK OFFICE
NOTICE OF RECORDATION OF ASSIGNMENT DOCUMENT

THE ENCLOSED DOCUMENT HAS BEEN RECORDED BY THE ASSIGNMENT DIVISION OF THE U.S. PATENT AND TRADEMARK OFFICE. A COMPLETE MICROFILM COPY IS AVAILABLE AT THE ASSIGNMENT SEARCH ROOM ON THE REEL AND FRAME NUMBER REFERENCED BELOW.

PLEASE REVIEW ALL INFORMATION CONTAINED ON THIS NOTICE. THE INFORMATION CONTAINED ON THIS RECORDATION NOTICE REFLECTS THE DATA PRESENT IN THE PATENT AND TRADEMARK ASSIGNMENT SYSTEM. IF YOU SHOULD FIND ANY ERRORS OR HAVE QUESTIONS CONCERNING THIS NOTICE, YOU MAY CONTACT THE EMPLOYEE WHOSE NAME APPEARS ON THIS NOTICE AT 703-308-9723. PLEASE SEND REQUEST FOR CORRECTION TO: U.S. PATENT AND TRADEMARK OFFICE, ASSIGNMENT DIVISION, BOX ASSIGNMENTS, NORTH TOWER BUILDING, SUITE 10C35, WASHINGTON, D.C. 20231.

RECORDATION DATE: 11/25/1996

REEL/FRAME: 8330/0684
NUMBER OF PAGES: 3

BRIEF: ASSIGNMENT OF ASSIGNOR'S INTEREST (SEE DOCUMENT FOR DETAILS).

ASSIGNOR:

SINGH, BRIJ P.

DOC DATE: 11/22/1996

ASSIGNOR:

ARORA, PRAMOD K.

DOC DATE: 11/22/1996

ASSIGNEE:

NANOFILM CORPORATION
10111 SWEET VALLEY DRIVE
VALLEY VIEW, OHIO 44125-4250

SERIAL NUMBER: 08755964

PATENT NUMBER:

FILING DATE: 11/25/1996

ISSUE DATE:

KIMBERLY BARNES, EXAMINER
ASSIGNMENT DIVISION
OFFICE OF PUBLIC RECORDS

RECEIVED

AUG 08 2001

OFFICE OF PETITIONS

02-07-1997

OMB No. 0651-0011 (Rev. 4/94)

495263010018

Tab settings: 0 0 0 0



08/755964

To the Honorable Commissioner of Patents

100351144

checked original documents or copy thereof.

1. Name of conveying party(ies):

Brij P. Singh and
Pramod K. AroraAdditional name(s) of conveying party(ies) attached? ☐ Yes ☒ No

2. Name and address of receiving party(ies)

Name: NanoFilm Corporation

Internal Address: 10111 Sweet Valley Drive

Valley View, Ohio 44125-4250

Street Address: (same as above)

City: State: ZIP:

Additional name(s) & address(es) attached? ☐ Yes ☒ No

3. Nature of conveyance: MRD 11-25-96

☒ Assignment☐ Merger☐ Security Agreement☐ Change of Name☐ Other

Execution Date: November 22, 1996

4. Application number(s) or patent number(s):

If this document is being filed together with a new application, the execution date of the application is: November 21, 1996

A. Patent Application No.(s)

B. Patent No.(s)

70189 U.S. PTO



02/05/97

Additional numbers attached? ☐ Yes ☒ No

5. Name and address of party to whom correspondence concerning document should be mailed:

Name: Barbara E. Arndt, Ph.D.

Internal Address: Jones, Day, Reavis & Pogue

North Point - 901 Lakeside Avenue

Cleveland, Ohio 44114-1190

Street Address: (same as above)

City: State: ZIP:

6. Total number of applications and patents involved: 1

7. Total fee (37 CFR 3.41).....\$ 40.00

☒ Enclosed☒ Authorized to be charged to deposit account for any deficit.

8. Deposit account number:

10-1202

(Attach duplicate copy of this page if paying by deposit account)

DO NOT USE THIS SPACE

40-E

9. Statement and signature.

To the best of my knowledge and belief, the foregoing information is true and correct and any attached copy is a true copy of the original document.

Barbara E. Arndt

Name of Person Signing

Signature

November 25, 1996

Date

Total number of pages including cover sheet, attachments, and document: 3

DOCKET NO. 495263010018

JOINT

A S S I G N M E N T

In consideration of One Dollar and other good and valuable consideration, receipt of which is hereby acknowledged, we, **BRIJ P. SINGH** and **PRAMOD K. ARORA**, ("ASSIGNORS"), citizens of the United States and India, respectively, residing in North Royalton, Ohio, respectively, hereby sell, transfer, set over and assign unto **NanoFilm Corporation** ("ASSIGNEE"), a corporation of the State of Ohio, having a principal place of business at 10111 Sweet Valley Drive, Valley View, Ohio 44125-4250, its successors, assigns, nominees, or other legal representatives, the entire right, title and interest in and to the invention **METHOD FOR MODIFYING SURFACES WITH ULTRA THIN FILMS** invented by us and the application for United States patent therefor, executed concurrently herewith, and all original and reissued patents granted therefor, and all divisions and continuations thereof, including the subject matter of any and all claims which may be obtained in every such patent, and the right to apply for and obtain patents in countries foreign to the United States, and in and to any Letters Patent which may be granted thereon in such foreign countries, and authorize and request the Commissioner of Patents and Trademarks of the United States, and any official of any country or countries foreign to the United States whose duty it is to issue patents on applications as aforesaid, to issue the said Letters Patent to the said ASSIGNEE, its successors, assigns, nominees or other legal representatives, as assignee and owner of the entire interest, and covenant that we have full right to convey the entire interest herein assigned and that we have not executed and will not execute any agreement in conflict herewith, and agree that we will communicate to said ASSIGNEE, its successors, assigns, nominees or other legal representatives, all facts known to us respecting said invention, whenever requested, and testify in any legal proceedings, sign all lawful papers, execute and deliver all divisional, continuing and reissue applications, make lawful papers, execute and deliver all divisional, continuing and reissue applications, make all rightful oaths and do all lawful acts requisite for the application for such divisional, continuing or reissue applications, or the procuring thereof, and that if and when said ASSIGNEE, its successors, assigns, nominees or other legal representatives desire to file a disclaimer relating thereto we will, upon request, sign and deliver all lawful papers requisite for the filing of such disclaimer, and we further covenant and agree that we will, at any time upon request, do everything legally possible to aid said ASSIGNEE, its successors, assigns, nominees or other legal representatives, either in its or our own

DOCKET NO. 495263010018

names, to apply for, obtain and enforce proper patent protection in all countries, including priority rights granted to patents in foreign countries according to all the laws and treaties in force, all without further consideration but at the expense of said ASSIGNEE, its successors, assigns, nominees or other legal representatives.



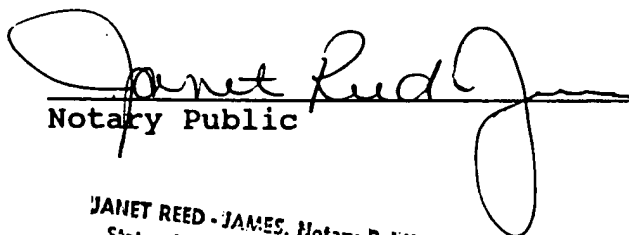
BRIJ P. SINGH



PRAMOD K. ARORA

STATE OF OHIO)
) SS.
COUNTY OF CUYAHOGA)

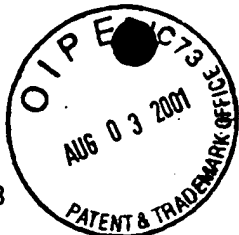
This 22 day of NOV, 1996 before me personally came the above-named BRIJ P. SINGH and PRAMOD K. ARORA, to me personally known as the individuals who executed the foregoing Assignment, and who acknowledged to me that they executed the same of their own free will for the purpose therein set forth.



Notary Public

(SEAL)

JANET REED-JAMES, Notary Public
State of Ohio, Cuyahoga County
My Commission Expires Nov. 5, 2000



UNITED STATES DEPARTMENT OF COMMERCE
Patent and Trademark Office
ASSISTANT SECRETARY AND COMMISSIONER
OF PATENTS AND TRADEMARKS
Washington, D.C. 20231

OCTOBER 01, 1998

PTAS

JONES, DAY, REAVIS & POGUE
JACQUELINE M. O'BRIEN
NORTH POINT
901 LAKESIDE AVENUE
CLEVELAND, OH 44114



100771999A

UNITED STATES PATENT AND TRADEMARK OFFICE
NOTICE OF RECORDATION OF ASSIGNMENT DOCUMENT

THE ENCLOSED DOCUMENT HAS BEEN RECORDED BY THE ASSIGNMENT DIVISION OF THE U.S. PATENT AND TRADEMARK OFFICE. A COMPLETE MICROFILM COPY IS AVAILABLE AT THE ASSIGNMENT SEARCH ROOM ON THE REEL AND FRAME NUMBER REFERENCED BELOW.

PLEASE REVIEW ALL INFORMATION CONTAINED ON THIS NOTICE. THE INFORMATION CONTAINED ON THIS RECORDATION NOTICE REFLECTS THE DATA PRESENT IN THE PATENT AND TRADEMARK ASSIGNMENT SYSTEM. IF YOU SHOULD FIND ANY ERRORS OR HAVE QUESTIONS CONCERNING THIS NOTICE, YOU MAY CONTACT THE EMPLOYEE WHOSE NAME APPEARS ON THIS NOTICE AT 703-308-9723. PLEASE SEND REQUEST FOR CORRECTION TO: U.S. PATENT AND TRADEMARK OFFICE, ASSIGNMENT DIVISION, BOX ASSIGNMENTS, CG-4, 1213 JEFFERSON DAVIS HWY, SUITE 320, WASHINGTON, D.C. 20231.

RECORDATION DATE: 07/20/1998

REEL/FRAME: 9322/0889
NUMBER OF PAGES: 3

BRIEF: ASSIGNMENT OF ASSIGNOR'S INTEREST (SEE DOCUMENT FOR DETAILS).

ASSIGNOR:
SINGH, BRIJ P.

DOC DATE: 07/07/1998

ASSIGNOR:
ARORA, PRAMOD K.

DOC DATE: 07/07/1998

ASSIGNEE:
NANOFILM, LTD.
10111 SWEET VALLEY DRIVE
VALLEY VIEW, OHIO 44125

SERIAL NUMBER: 09084944
PATENT NUMBER:

FILING DATE: 05/26/1998
ISSUE DATE:

JEEVON JONES, EXAMINER
ASSIGNMENT DIVISION
OFFICE OF PUBLIC RECORDS

RECEIVED

AUG 08 2001

OFFICE OF PETITIONS

RE



100771999

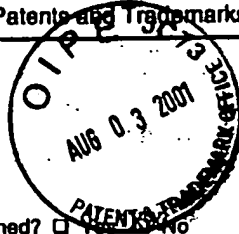
Tab settings ☐ ☐ ☐ ☐

To the Honorable Commissioner of Patents and Trademarks: Please record the attached original documents or copy thereof.

1. Name of conveying party(ies):

Brij P. Singh
Pramod K. Arora

Additional name(s) of conveying party(ies) attached? ☐ Yes ☒ No



2. Name and address of receiving party(ies)

Name: nanoFILM, Ltd.

Internal Address:

Street Address: 10111 Sweet Valley Drive

City: Valley View State: OH ZIP: 44125

Additional name(s) & address(es) attached? ☐ Yes ☒ No

3. Nature of conveyance:

☒ Assignment

☐ Merger

☐ Security Agreement

☐ Change of Name

☐ Other

Execution Date: July 7, 1998

4. Application number(s) or patent number(s):

If this document is being filed together with a new application, the execution date of the application is:

A. Patent Application No.(s)

09/084,944 filed May 26, 1998

B. Patent No.(s)

AUG 08 2001

OFFICE OF PETITIONS

Additional numbers attached? ☐ Yes ☒ No

RECEIVED

5. Name and address of party to whom correspondence concerning document should be mailed:

Name: Jacqueline M. O'Brien

Internal Address: JONES, DAY, REAVIS & POGUE
North Point

Street Address: 901 Lakeside Avenue

City: Cleveland State: OH ZIP: 44114

6. Total number of applications and patents involved: 1

7. Total fee (37 CFR 3.41).....\$ 40.00

☒ Enclosed

☒ Authorized to be charged to deposit account ,
if over or underpayment

8. Deposit account number:

10-1202

(Attach duplicate copy of this page if paying by deposit account)

DO NOT USE THIS SPACE

9. Statement and signature.

To the best of my knowledge and belief, the foregoing information is true and correct and any attached copy is a true copy of the original document.

Jacqueline M. O'Brien

Name of Person Signing

Jacqueline M. O'Brien

Signature

July 15, 1998

Date

Total number of pages including cover sheet, attachments, and document: 3

Mail documents to be recorded with required cover sheet information to:
Commissioner of Patents & Trademarks, Box Assignments
Washington, D.C. 20231

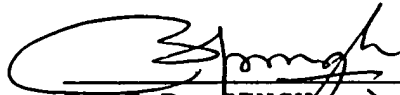
JOINT

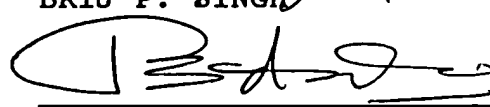
A S S I G N M E N T

In consideration of One Dollar and other good and valuable consideration, receipt of which is hereby acknowledged, we, **BRIJ P. SINGH** and **PRAMOD K. ARORA**, ("ASSIGNORS"), citizens of the United States and India, respectively, residing in North Royalton, Ohio, respectively, hereby sell, transfer, set over and assign unto **nanofilm, Ltd.** ("ASSIGNEE"), an Ohio Limited Liability Company, having a principal place of business at 10111 Sweet Valley Drive, Valley View, Ohio 44125-4250, its successors, assigns, nominees, or other legal representatives, the entire right, title and interest in and to the invention **METHOD FOR MODIFYING SURFACES WITH ULTRA THIN FILMS** invented by us and United States Patent Application Serial No. 09/084,944, filed May 26, 1998 therefor, and all original and reissued patents granted therefor, and all divisions and continuations thereof, including the subject matter of any and all claims which may be obtained in every such patent, and the right to apply for and obtain patents in countries foreign to the United States, and in and to any Letters Patent which may be granted thereon in such foreign countries, and authorize and request the Commissioner of Patents and Trademarks of the United States, and any official of any country or countries foreign to the United States whose duty it is to issue patents on applications as aforesaid, to issue the said Letters Patent to the said ASSIGNEE, its successors, assigns, nominees or other legal representatives, as assignee and owner of the entire interest, and covenant that we have full right to convey the entire interest herein assigned and that we have not executed and will not execute any agreement in conflict herewith, and agree that we will communicate to said ASSIGNEE, its successors, assigns, nominees or other legal representatives, all facts known to us respecting said invention, whenever requested, and testify in any legal proceedings, sign all lawful papers, execute and deliver all divisional, continuing and reissue applications, make lawful papers, execute and deliver all divisional, continuing and reissue applications, make all rightful oaths and do all lawful acts requisite for the application for such divisional, continuing or reissue applications, or the procuring thereof, and that if and when said ASSIGNEE, its successors, assigns, nominees or other legal representatives desire to file a disclaimer relating thereto we will, upon request, sign and deliver all lawful papers requisite for the filing of such disclaimer, and we further covenant and agree that we will, at any time upon request, do everything legally possible to aid said ASSIGNEE, its successors, assigns, nominees or other legal representatives, either in its or our own

DOCKET NO. 495263010019

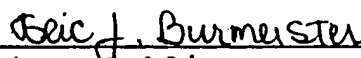
names, to apply for, obtain and enforce proper patent protection in all countries, including priority rights granted to patents in foreign countries according to all the laws and treaties in force, all without further consideration but at the expense of said ASSIGNEE, its successors, assigns, nominees or other legal representatives.


BRIJ P. SINGH


PRAMOD K. ARORA

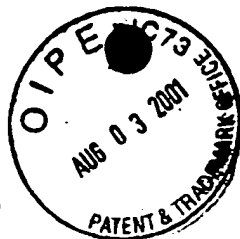
STATE OF OHIO)
) SS.
COUNTY OF CUYAHOGA)

This 7TH day of July, 1998 before me personally came the above-named BRIJ P. SINGH and PRAMOD K. ARORA, to me personally known as the individuals who executed the foregoing Assignment, and who acknowledged to me that they executed the same of their own free will for the purpose therein set forth.


Notary Public

ERIC BURMEISTER, Notary Public
State of Ohio, Cuyahoga County
My Commission Expires Oct. 7, 2002

(SEAL)



**UNITED STATES DEPARTMENT OF COMMERCE
Patent and Trademark Office**

ASSISTANT SECRETARY AND COMMISSIONER
OF PATENTS AND TRADEMARKS
Washington, D.C. 20231

FEBRUARY 02, 1999

PTAS

JONES, DAY, REAVIS & POGUE
H. DUANE SWITZER
NORTH POINT
901 LAKESIDE AVENUE
CLEVELAND, OHIO 44114



100850569A

**UNITED STATES PATENT AND TRADEMARK OFFICE
NOTICE OF RECORDATION OF ASSIGNMENT DOCUMENT**

THE ENCLOSED DOCUMENT HAS BEEN RECORDED BY THE ASSIGNMENT DIVISION OF THE U.S. PATENT AND TRADEMARK OFFICE. A COMPLETE MICROFILM COPY IS AVAILABLE AT THE ASSIGNMENT SEARCH ROOM ON THE REEL AND FRAME NUMBER REFERENCED BELOW.

PLEASE REVIEW ALL INFORMATION CONTAINED ON THIS NOTICE. THE INFORMATION CONTAINED ON THIS RECORDATION NOTICE REFLECTS THE DATA PRESENT IN THE PATENT AND TRADEMARK ASSIGNMENT SYSTEM. IF YOU SHOULD FIND ANY ERRORS OR HAVE QUESTIONS CONCERNING THIS NOTICE, YOU MAY CONTACT THE EMPLOYEE WHOSE NAME APPEARS ON THIS NOTICE AT 703-308-9723. PLEASE SEND REQUEST FOR CORRECTION TO: U.S. PATENT AND TRADEMARK OFFICE, ASSIGNMENT DIVISION, BOX ASSIGNMENTS, CG-4, 1213 JEFFERSON DAVIS HWY, SUITE 320, WASHINGTON, D.C. 20231.

RECORDATION DATE: 10/01/1998

REEL/FRAME: 9498/0980
NUMBER OF PAGES: 3

BRIEF: ASSIGNMENT OF ASSIGNOR'S INTEREST (SEE DOCUMENT FOR DETAILS).

ASSIGNOR:

SINGH, BRIJ P.

DOC DATE: 10/01/1998

ASSIGNOR:

ARORA, PRAMOD K.

DOC DATE: 10/01/1998

ASSIGNEE:

NANOFILM, LTD.
10111 SWEET VALLEY DRIVE
VALLEY VIEW, OHIO 44125

SERIAL NUMBER: 09164489
PATENT NUMBER:

FILING DATE: 10/01/1998
ISSUE DATE:

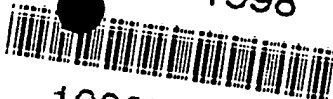
RECEIVED

KIMBERLY WHITE, EXAMINER
ASSIGNMENT DIVISION
OFFICE OF PUBLIC RECORDS

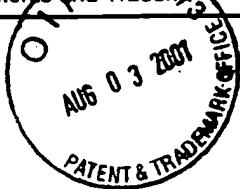
AUG 08 2001

OFFICE OF PETITIONS

10-14-1998



100850560

VER SHE
LYU.S. DEPARTMENT OF COMMERCE
Patent and Trademark Office

To the Honorable Commissioner of Patents and Trademarks

and the attached original documents or copy thereof.

1. Name of conveying party(ies):

Brij P. Singh and
Pramod K. AroraAdditional name(s) of conveying party(ies) attached? ☐ Yes ☐ No

3. Nature of conveyance:

☒ Assignment☐ Merger☐ Security Agreement☐ Change of Name☐ Other _____Execution Date: October 1, 1998

2. Name and address of receiving party(ies)

Name: nanoFILM, Ltd.

Internal Address: _____

Street Address: 10111 Sweet Valley DriveCity: Valley View State: Ohio ZIP: 44125Additional name(s) & address(es) attached? ☐ Yes ☒ No

4. Application number(s) or patent number(s):

If this document is being filed together with a new application, the execution date of the application is: Sept. 30, 1998

A. Patent Application No.(s)

B. Patent No.(s)

RECEIVED

AUG 08 2001

OFFICE OF PETITIONS

Additional numbers attached? ☐ Yes ☒ No

5. Name and address of party to whom correspondence concerning document should be mailed:

Name: H. Duane SwitzerInternal Address: Jones, Day, Reavis & PoqueStreet Address: North Point901 Lakeside AvenueCity: Cleveland State: Ohio ZIP: 441146. Total number of applications and patents involved: 17. Total fee (37 CFR 3.41).....\$ 40.00☒ Enclosed☒ Authorized to be charged to deposit account
for any deficit.

8. Deposit account number:

10-1202

(Attach duplicate copy of this page if paying by deposit account)

9. Statement and signature.

To the best of my knowledge and belief, the foregoing information is true and correct and any attached copy is a true copy of the original document.

H. Duane Switzer

Name of Person Signing

H. Duane Switzer
SignatureOctober 1, 1998

Date

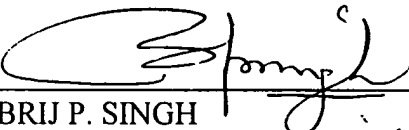
Total number of pages including cover sheet, attachments, and document: 3

ASSIGNMENT

In consideration of One Dollar and other good and valuable consideration, receipt of which is hereby acknowledged, we, **BRIJ P. SINGH** and **PRAMOD K. ARORA**, ("ASSIGNORS"), citizens of the United States and India, respectively, residing in North Royalton, Ohio, respectively, hereby sell, transfer, set over and assign unto **nanoFILM, Ltd.** ("ASSIGNEE"), an Ohio Limited Liability Company of the State of Ohio, having a principal place of business at 10111 Sweet Valley Drive, Valley View, Ohio 44125-4250, its successors, assigns, nominees, or other legal representatives, the entire right, title and interest in and to the invention **HYDROPHOBIC THIN FILMS ON MAGNESIUM FLUORIDE SURFACES** invented by us and the application for United States patent therefor, executed concurrently herewith, and all original and reissued patents granted therefor, and all divisions and continuations thereof, including the subject matter of any and all claims which may be obtained in every such patent, and the right to apply for and obtain patents in countries foreign to the United States, and in and to any Letters Patent which may be granted thereon in such foreign countries, and authorize and request the Commissioner of Patents and Trademarks of the United States, and any official of any country or countries foreign to the United States whose duty it is to issue patents on applications as aforesaid, to issue the said Letters Patent to the said ASSIGNEE, its successors, assigns, nominees or other legal representatives, as assignee and owner of the entire interest, and covenant that we have full right to convey the entire interest herein assigned and that we have not executed and will not execute any agreement in conflict herewith, and agree that we will communicate to said ASSIGNEE, its successors, assigns, nominees or other legal representatives, all facts known to us respecting said invention, whenever requested, and testify in any legal proceedings, sign all lawful papers, execute and deliver all divisional, continuing and reissue applications, make lawful papers, execute and deliver all divisional, continuing and reissue applications, make all rightful oaths and do all lawful acts requisite for the application for such divisional, continuing or reissue applications, or the procuring thereof, and that if and when said ASSIGNEE, its successors, assigns, nominees or other legal representatives desire to file a disclaimer relating thereto we will, upon request, sign and deliver all lawful papers requisite for the filing of such disclaimer, and we further covenant and agree that we will, at any time upon request, do everything legally possible to aid said ASSIGNEE, its successors, assigns, nominees or other legal representatives, either in its or our own names, to apply for, obtain and enforce proper patent protection in all countries, including priority rights granted to patents in foreign countries according to all the laws and treaties in force, all without further consideration but at

DOCKET NO. 495263010020

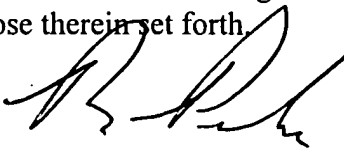
the expense of said ASSIGNEE, its successors, assigns, nominees or other legal representatives.


BRIJ P. SINGH


PRAMOD K. ARORA

STATE OF OHIO)
) SS.
COUNTY OF CUYAHOGA)

This 1ST day of October, 1998 before me personally came the above-named BRIJ P. SINGH and PRAMOD K. ARORA, to me personally known as the individuals who executed the foregoing Assignment, and who acknowledged to me that they executed the same of their own free will for the purpose therein set forth.



Notary Public

(SEAL)

REI 07-15-1998

SHEET

U.S. DEPARTMENT OF COMMERCE
Patent and Trademark Office

Tab settings 0000 ▼

To the Honorable Commissioner of Patents

00764414

attached original documents or copy thereof.

1. Name of conveying party(ies):

NanoFilm Corporation

Additional name(s) of conveying party(ies) attached? ☐ Yes ☒ No

2. Name and address of receiving party(ies)

Name: nanoFILM, Ltd.

Internal Address:

Street Address: 10111 Sweet Valley Drive

City: Valley View State: Ohio ZIP: 44125

Additional name(s) & address(es) attached? ☐ Yes ☒ No

3. Nature of conveyance:

☐ Assignment

☒ Merger

☐ Security Agreement

☐ Change of Name

☐ Other

Execution Date: June 30, 1995

4. Application number(s) or patent number(s):

If this document is being filed together with a new application, the execution date of the application is: _____

A. Patent Application No.(s)

See Attachment A

B. Patent No.(s)

See Attachment A

Additional numbers attached? ☒ Yes ☐ No

5. Name and address of party to whom correspondence concerning document should be mailed:

Name: Jacqueline M. O'Brien

Internal Address: JONES DAY REAVIS & POGUE

North Point

Street Address: 901 Lakeside Avenue

City: Cleveland State: Ohio ZIP: 44114

6. Total number of applications and patents involved: 11

7. Total fee (37 CFR 3.41).....\$ 440.00 E

☒ Enclosed

☒ Authorized to be charged to deposit account, if over or underpayment

8. Deposit account number:

10-1202

(Attach duplicate copy of this page if paying by deposit account)

07/14/1998 JTS/BAZZ 00000106 5076791

DO NOT USE THIS SPACE

01-FC-881

440.00.00

9. Statement and signature.

To the best of my knowledge and belief, the foregoing information is true and correct and any attached copy is a true copy of the original document.

Jacqueline M. O'Brien

Name of Person Signing

Jacqueline M. O'Brien

Signature

6/30/98

Date

Total number of pages including cover sheet, attachments, and document: 9

Mail documents to be recorded with required cover sheet information to:
Commissioner of Patents & Trademarks, Box 2023
Washington, D.C. 20231

REEL: 9297 FRAME: 0701

ATTACHMENT A

<u>U.S. Patent No.</u>	<u>Title</u>	<u>Issue Date</u>
5,078,791	Film Forming Composition and Method for Modifying Surfaces with Ultra-Thin Films	01/07/92
5,106,561	Ultra-Thin Molecular Film	04/21/92
5,166,000	Solution Containing Amphiphilic Molecules	11/24/92
5,173,365	Ultra-Thin Molecular Film	12/22/92
5,204,126	Thin Films	04/20/93
5,219,654	Film Forming Composition and Method for Modifying Surfaces with Ultra-Thin Films	06/15/93
5,300,561	Solution Containing Amphiphilic Molecules	04/05/94
5,766,698	Method for Modifying Surfaces with Ultra Thin Films	06/16/98

<u>U.S. Patent Application No.</u>	<u>Title</u>	<u>Filing Date</u>
08/321,088	Ultra-Thin Molecular Film	03/09/89
08/992,146	Method for Modifying Surfaces with Ultra Thin Films	12/17/97
09/084,944	Method for Modifying Surfaces with Ultra Thin Films	05/26/98

**Canadian Patent
Application No.**

Title

Filing Date

2,120,252	Solution Containing Amphiphilic Molecules	03/29/94
2,217,576	Method for Modifying Surfaces with Ultra Thin Films	10/07/97

**European Patent
Application No.**

92906253.7	Thin Films	11/20/91
92918168.3	Solution Containing Amphiphilic Molecules	08/12/92
97202777.5	Thin Films	09/10/97
97309401.4	Method for Modifying Surfaces with Ultra Thin Films	11/21/97

**Japanese Patent
Application No.**

297244/89	Ultra-Thin Molecular Film	11/15/89
-----------	---------------------------	----------

**Patent Cooperation
Treaty Application No.**

PCT/US91/08748	Thin Films	11/20/91
----------------	------------	----------



Prescribed by
Bob Taft, Secretary of State
30 East Broad Street, 14th Floor
Columbus, Ohio 43266-0418
Form MER (July 1994)

Approved _____
Date _____
Fee _____

CERTIFICATE OF MERGER

In accordance with the requirements of Ohio law, the undersigned corporations, limited liability companies and/or limited partnerships, desiring to effect a merger, set forth the following facts:

I. SURVIVING ENTITY

A. The name of the entity surviving the merger is:

nanoFILM, Ltd.

(If the surviving entity is an Ohio limited partnership or qualified foreign limited partnership, its registration number must be provided)

B. Name change: As a result of this merger, the name of the surviving entity has been changed to the following: _____

only if the name of surviving entity is changing through the merger

C. The surviving entity is a: *(Please check the appropriate box and fill in the appropriate blanks)*

☐ Domestic (Ohio) corporation

☐ Foreign (Non-Ohio) corporation incorporated under the laws of the state/ country of _____ and licensed to transact business in the state of Ohio.

☐ Foreign (Non-Ohio) corporation incorporated under the laws of the state/country of _____, and NOT licensed to transact business in the state of Ohio.

~~xxxxxx~~ Domestic (Ohio) limited liability company

☐ Foreign (Non-Ohio) limited liability company organized under the laws of the state/country of _____, and registered to do business in the state of Ohio.

☐ Foreign (Non-Ohio) limited liability company organized under the laws of the state/country of _____, and NOT registered to do business in the state of Ohio.

☐ Domestic (Ohio) limited partnership, registration number _____

- [] Foreign (Non-Ohio) limited partnership organized under the laws of the state/country of _____, and registered to do business in the state of Ohio, under registration number _____
- [] Foreign (Non-Ohio) limited partnership organized under the laws of the state/country of _____, and NOT registered to do business in the state of Ohio.

II. Merging Entities

The name, type of entity, and state/country of incorporation or organization, respectively, of each entity, other than the survivor, which is a party to the merger are as follows: (if transferee entity to cover this form, please attach a separate sheet listing the merging entities. Ohio registered or foreign qualified limited partnerships must include registration number)

Name	State/ Country of Organization	Type of Entity
NanoFilm Corporation	Ohio	corporation
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

III. Merger Agreement on File

The name and mailing address of the person or entity from whom/which eligible persons may obtain a copy of the agreement of merger upon written request:

Name	Address
Scott E. Rickert	10111 Sweet Valley Drive
	(street and number)
	Valley View, OH 44125-4250
	(city, village or township) (state) (zip code)

IV. Effective Date of Merger

This merger is to be effective:

On June 30, 1995 (if a date is specified, the date must be a date on or after the date of filing; the effective date of the merger cannot be earlier than the date of filing; if no date is specified, the date of filing will be the effective date of the merger).

The laws of the state or country under which each constituent entity exists, permits this merger.

This merger was adopted, approved and authorized by each of the constituent entities in compliance with the laws of the state under which it is organized, and the persons signing this certificate on behalf of each of the constituent entities are duly authorized to do so.

VI. Statutory Agent

Name**Address**

A.G.C. Co.

1900 East Ninth Street, Suite 3200

(complete street address)

Cleveland, OH 44114

(city, village or township)

(29 words)

(This item MUST be completed if the surviving entity is a foreign entity which is not licensed, registered or otherwise authorized to conduct or transact business in the State of Ohio)

Acceptance of Agent

The undersigned, named herein as the statutory agent for the above referenced surviving entity, hereby acknowledges and accepts the appointment of statutory agent for said entity.

Signature of Agent

(The acceptance of agent must be completed by domestic surviving entities if through this merger the statutory agent for the surviving entity has changed, or the named agent differs in any way from the name reflected on the Secretary of State's records.)

VII. Statement of Merger

Upon filing, or upon such later date as specified herein, the merging entity/entities listed herein shall merge into the listed surviving entity.

VIII. Amendments

The articles of incorporation, articles of organization or certificate of limited partnership (strike the inapplicable terms) of the surviving domestic entity herein, are amended as set forth in the attached "Exhibit A"

(Please note that any amendments to articles of incorporation, articles of organization or to a certificate of limited partnership MUST be attached if the surviving entity is a DOMESTIC corporation, limited liability company, or limited partnership.)

CERTIFICATION

I hereby certify that the attached Certificate of Merger of NanoFilm Corporation
into nanoFILM, Ltd., an Ohio Limited Liability Company, is a true and exact copy of the original.

STATE OF OHIO)
) ss.
COUNTY OF CUYAHOGA)

Jacqueline M O'Brien
Jacqueline M. O'Brien - Notary Public

Date: June 30, 1998

My Commission Expires:

JACQUELINE M. O'BRIEN
Notary Public, State of Ohio, Cuy. Cty.
My Commission Expires Aug. 2, 1999



Creation date: 01-08-2004
Indexing Officer: VHARRIS - VAUGHN HARRIS
Team: OIPEBackFileIndexing
Dossier: 09887661

Legal Date: 10-09-2001

No.	Doccode	Number of pages
1	CTMS	1

Total number of pages: 1

Remarks:

Order of re-scan issued on